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SPECIFICATION FOR MINERAL OIL, FOOD GRADE

UDC 664·022·3 : 665·7·03

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Indian Standard

SPECIFICATION FOR MINERAL OIL, FOOD GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 4 April 1983, after the draft finalized by the Food Additives Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Use of mineral oil, food grade is being permitted as lubricant for certain food products in the Prevention of Food Adulteration Rules, 1955.

0.3 In the preparation of this standard considerable assistance has been derived from:

a) Specification for identity and purity of some food additives, FAO Nutrition Meeting Report Series No. 57, FAO/WHO, Rome 1977; and

b) Food Chemicals Codex, ED 1, 1966 Published by NAS — NRC, Washington DC.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard specifies the requirements and methods of sampling and test for mineral oil, food grade.

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*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — Mineral oil, food grade is a mixture of liquid hydrocarbons, essentially parafinic and napthenic in nature, obtained from petroleum, refined by the use of oleum. This shall exclude mineral oils produced by the hydrogenation process unless they have been subsequently refined by the use of oleum and exclude other types of white mineral oils to which antioxidants may have been added for technological purposes.

2.1.1 Mineral oil, food grade is a colourless, transparent and oily liquid and free from fluorescence. It is odourless and tasteless.

2.2 Identification

2.2.1 *Solubility* — Insoluble in water and ethanol, soluble in volatile oils, miscible with fixed oils.

2.2.2 The product burns with bright flame and with paraffine like characteristic offensive smell.

2.2.3 When heated with sulphur, it produces hydrogen sulphide like smell.

2.3 Specific Gravity — shall be between 0·830 and 0·905.

2.4 Petroleum — When heated on water bath, the product shall not produce petroleum-like smell.

2.5 The material shall conform to the requirements given in Table 1.

3. PACKING, STORAGE AND MARKING

3.1 Packing — The material shall be filled in containers with as little air space as possible. The containers shall be such as to preclude air contamination of the contents with metals or other impurities.

3.2 Storage — The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

3.3 Marking — Each container shall be marked legibly to give the following information:

a) Name of the material including the words 'Food Grade',
b) Name and address of the manufacturer,
c) Minimum net mass of contents,
d) Batch or code number, and
e) Date of manufacture.

TABLE 1 REQUIREMENTS FOR MINERAL OIL FOOD GRADE

(*Clauses* 2.5 and 5.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix A	Cl No. in IS : 1699-1974*
(1)	(2)	(3)	(4)	(5)
i)	Acidity or alkalinity	Passes the test	A-1	—
ii)	Readily carbonizable substances	do	A-2	—
iii)	Polynuclear aromatic hydrocarbons, absorbance at wave lengths between 260-350 nm, *Max*	0·10	A-3	—
iv)	Solid paraffins	Passes the test	A-4	—
v)	Heavy metals, mg/kg, *Max*	10	A-5	—
vi)	Lead (as Pb), mg/kg, *Max*	1	—	9
vii)	Arsenic (as As), mg/kg, *Max*	1	—	10
viii)	Sulphurs (as SO_4)	Passes the test	A-6	—

*Methods of sampling and test for food colours (*first revision*).

3.3.1 The container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in Appendix A of IS : 2557-1963*.

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*Specification for annatto colour for food products.

5. TESTS

5.1 Tests shall be carried out by the methods specified in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1)

METHOD OF TEST FOR MINERAL OIL, FOOD GRADE

A-1. ACIDITY OR ALKALINITY

A-1.1 Add about 10 ml of hot water and a drop of phenolphthalein TS to 10 ml of the sample and shake vigorously. The solution does not turn red. Furthermore, add about 0·2 ml of 0·02 N sodium hydroxide to the mixture and shake it. The mixture turns red.

A-2. READILY CARBONIZABLE SUBSTANCES

A-2.1 Reagents

A-2.1.1 *Sulpuric Acid TS* — Add a quantity of sulpuric acid of known concentration to sufficient water to adjust the final concentration to between 94·5 and 95·5 percent of H_2SO_4. Since the acid concentration may change upon standing or upon intermittent use, the concentration should be checked frequently and solutions assaying more than 95·5 percent or less than 94·5 percent discarded or adjusted by adding either diluted or fuming sulphuric acid, as required.

A-2.1.2 *Cobaltous Chloride C.S.* — Dissolve about 65 g of cobaltous chloride ($CoCl_2.6H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 5 ml of this solution into a 250-ml iodine flask, add 5 ml hydrogen peroxide TS and 15 ml of sodium hydroxide solution (1 in 5), boil for 10 minutes, cool, and add 2 g of potassium iodide and 20 ml of dilute sulphuric acid (1 in 4). When the precipitate has dissolved, titrate the liberated iodine with 0·1 *N* sodium thiosulphate. The titration is sensitive to air oxidation and should be blanketed with carbon dioxide. Each ml of

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*Specification for water for general laboratory use (*second revision*).

0·1 *N* sodium thiosulphate is equivalent to 23·79 mg of $CoCl_2.6H_2O$. Adjust the final volume of the solution by the addition of enough of the mixture of hydrochloric acid and water to make each ml contain 59·5 mg of $CoCl_2.6H_2O$.

A-2.1.3 *Cupric Sulphate C.S.* — Dissolve about 65 g of cupric sulphate ($CuSO_4.5H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 10 ml of this solution into a 250-ml iodine flask, add 40 ml of water, 4 ml of acetic acid, and 3 g of potassium iodide, and titrate the liberated iodine with 0·1 *N* sodium thiosulphate, adding starch TS as the indicator. Each ml of 0·1 *N* sodium thiosulphate is equivalent to 24·97 mg of $CuSO_4.5H_2O$. Adjust the final volume of the solution by the addition of enough of the mixture of hydrochloric acid and water to make each ml contain 62·4 mg of $CuSO_4.5H_2O$.

A-2.1.4 *Ferric Chloride C.S.* — Dissolve about 55 g of ferric chloride ($FeCl_3.6H_2O$) in enough of mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 10 ml of this solution into a 250-ml iodine flask, add 15 ml of water, 5 ml of hydrochloric acid, and 3 g of potassium iodide, and allow the mixture to stand for 15 minutes. Dilute with 100 ml of water, and titrate the liberated iodine with 0·1 *N* sodium thiosulphate, adding starch TS as the indicator. Perform a blank determination with the same quantities of the same reagents and in the same manner and make any necessary correction. Each ml of 0·1 *N* sodium thiosulphate is equivalent to 27·03 mg of $FeCl_3.6H_2O$. Adjust the final volume of the solution by addition of the mixture of hydrochloric acid and water to make each ml contain 45·0 mg of $FeCl_3.6H_2O$.

A-2.2 Procedure

A-2.2.1 Place 5 ml of the sample into a cleaned and dried glass stoppered test tube, add 5 ml of 94·5 to 94·9 percent sulphuric acid. Shake up and down vigorously for 5 seconds at intervals of 2 minutes each while heating for 10 minutes on a water-bath. White mineral oil does not change colour and the colour of the sulphuric acid layer is not darker than that of a standard colour produced by mixing in a similar tube 3 ml of ferric chloride TSC, 1·5 ml of cobaltous chloride TSC and 0·5 ml of cupric sulphate TSC, this mixture being overlaid with 5 ml of mineral oil.

A-3. POLYNUCLEAR AROMATIC HYDROCARBONS

A-3.1 Take 25 ml of the sample with measuring cylinder of 25 ml volume, and transfer into separating funnel of 100 ml volume. Take 25 ml of *n*-hexane with same measuring cylinder and transfer into the separating funnel, and shake well. Add 5 ml of dimethyl sulphoxide and shake vigorously for 2 minutes and then allow to stand for 2 minutes. Transfer the separated lower layer into separating funnel of 50 ml volume, add 2 ml of *n*-hexane, shake vigorously for 2 minutes, and then allow to stand for 2 minutes. Transfer the lower layer into stopperable centrifugal tube and centrifuge for 10 minutes at 2 500 to 3 000 rev/min. Transfer the clear solution thus obtained, into cuvette of 1 cm path-length, stopper and use as sample.

Separately, take 25 ml of *n*-hexane into separating funnel of 50 ml volume, add 5 ml of dimethylsulphoxide and shake for 2 minutes vigorously and then allow to stand for 2 minutes. Transfer the separated lower layer into stopperable centrifugal tube of 10 ml volume and centrifuge for 10 minutes at 2 500 to 3 000 rev/min. Clear solution thus obtained is transferred into cuvette of 1 cm path-length, stopper and use as the control. Determine the absorbance of the sample solution, which should not exceed 0·1 at wavelength of between 260 to 350 nm. Use both *n*-hexane and dimethylsulphoxide in quality specified for ultraviolet spectrophotometry.

A-4. SOLID PARAFFINS

A-4.1 Place a reasonable quantity of the sample previously dried by heating at 100°C for two hours and cooled in a desiccator over sulphuric acid, in a glass cylindrical vessel having an internal diameter of approximate 25 mm. Close the vessel and immerse in a mixture of ice and water; after four hours the liquid is sufficiently clear so that a black line, 0·5 mm in width, held vertically behind the vessel, is easily seen.

A-5. HEAVY METALS

A-5-1 Reagents

A-5.1.1 *Ammonia Solution* — Dilute 400 ml of ammonium hydroxide (28 percent) to 1 000 ml with water.

A-5.1.2 *Hydrochloric Acid* — 10 percent.

A-5.1.3 *Lead Nitrate Stock Solution* — Dissolve 159·8 mg of lead nitrate in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1 000 ml and mix. Prepare and store the solution in lead-free glass containers.

A-5.1.4 *Standard Lead Solution* — Dilute 10 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of 10 μg of lead ion (Pb). Prepare the solution on the day of use.

A-5.1.5 *Nitric Acid* — 10 percent.

A-5.1.6 *Sulphuric Acid* — 94·5 to 95·5 percent.

A-5.1.7 *Acetic Acid* — 6 percent.

A-5.1.8 *Hydrogen Sulphide* — A saturated solution of hydrogen sulphide made by passing through H_2S in cold water.

A-5.2 Procedure

A-5.2.1 *Solution A*

Take 2 ml of the standard lead solution in 50 ml Nessler tube and add 23 ml of water. Adjust the *p*H to between 3·0 to 4·0 by addition of acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

A-5.2.2 *Solution B*

Place 2 g of the sample, accurately weighed in a suitable crucible, heat carefully until thoroughly charred. Then ignite, preferably in a muffle furnace, at 500 to 600°C until the carbon is all burned off. Cool, add 4 ml of dilute hydrochloric acid, cover and digest on a steam bath for 10 to 15 minutes. Uncover, and slowly evaporate on a steam-bath to dryness. Add 2 ml of dilute acetic acid and dilute with water to 25 ml. Wash the crucible and the filter with 10 ml of water. Transfer to a 50 ml Nessler tube. Dilute the combined filterate and washing with water to 40 ml and mix.

A-5.2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide mix and allow to stand for 45 minutes and view down over a white surface. The colour of Solution B shall not be darker than that of Solution A.

A-6. SULPHUR (as SO_4)

A-6.1 To 1 ml of a 25·0 percent *w/v* solution of barium chloride add 1·5 ml of ethanol sulphate standard solution (10 ppm SO_4), shake and allow to stand for one minute. Add 15 ml of the prescribed solution or a solution of the specified quantity of substance being examined in 15 ml of water and 0·5 ml of 5 M acetic acid. After five minutes any opalescence produced is not more intense than that of a standard prepared in a similar manner using 15 ml of sulphate standard solution (10 ppm SO_4) in place of the solution being examined.

(*Continued from page* 2)

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